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ULTRASONIC CHARACTERIZATION OF PLASMA SPRAY COATING

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INTRODUCTION

Plasma-spray coatings are widely used in industry to protect the substrate against aggressive environments or to alter the surface properties to enhance the wear resistance[1]. The coating is an aggregate of powder particles which are impacted after being heated on the substrate surface. As a result of the fast cooling rates [2], no significant diffusion occurs at the coating/substrate interface and between subsequent coating layers. Different defects in the coating layer often occur, such as delaminations between subsequent deposition layers, pores, non-melted particles, cracks due to thermal coefficient mismatch and loss of adhesion between coatings and substrates.

It is therefore increasingly important to be able to assess the properties of the coating layer and in particular the quality of the adhesion between the coating and the substrate. The standard method of evaluating coatings is destructive, namely cutting a sample and examining the cross section. The adhesion is measured as described in ASTM C 633[1] on a sample made from two coated cylinders which are bonded together forming a butt joint. The tensile test is performed perpendicular to the bonded sandwich surface and is limited by the strength of the adhesive bond between the two coatings.

In this work, an ultrasonic method for measuring the elastic properties of ceramic plasma-sprayed coatings is described. The ultrasonic measurements are complicated by very strong attenuation which occurs due to extreme microstructural inhomogeneity of the coating. Several techniques which are described below were used to measure the properties of the coating and to retrieve information on its adhesion to the substrate.

PLASMA SPRAYED SAMPLES

In this work, ceramic plasma-sprayed coatings were investigated. A 0.47 millimeters Cr_2O_3 coating deposited on a 2.12 millimeters plain steel substrate was studied. A piece of the specimen was cut off for optical and electronic microscopy studies. From the remaining sample, another part was cut off and the substrate was dissolved in acid (50% HNO_3-50% H_2O) to measure the properties of the plasma sprayed coating. The coated substrate remaining for ultrasonic measurements was 55x25 millimeters. All

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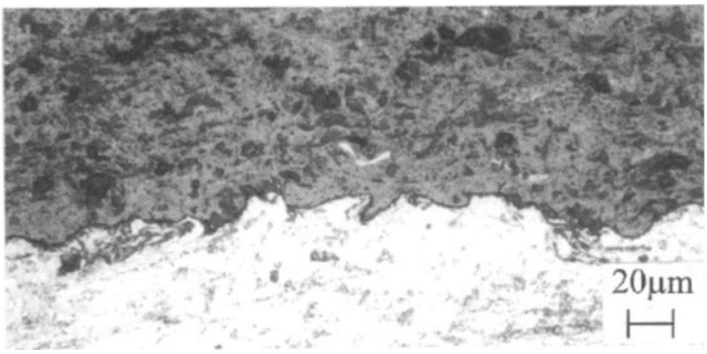

Figure 1. Optical micrograph of the microstructure of the Cr_2O_3-steel interface, unetched.

the ultrasonic measurements were conducted in water at a constant temperature of 29.8±0.01°C.

The micrograph of the Cr_2O_3 plasma-sprayed specimen shown in figure 1 reveals a typical microstructure. The coating is laminar in nature and is highly non-homogeneous; it contains pores, some non-melted powder particles (round in shape) and second phase (bright and elongated) particles which are dispersed throughout the layer.

Good metallurgical contact can be observed in this sample between the coating and the substrate. The coating is well attached to the substrate's rough surface forming good mechanical interconnection.

The density of the Cr_2O_3 coating was determined using the water displacement method. It is 4.85gr/cm^3 which is 93% of the theoretical density of Cr_2O_3 (5.21gr/cm^3[3]).

ULTRASONIC MEASUREMENTS

In-Plane Elastic Moduli of the Coating

The plate mode anti-resonance technique was used to determine the in-plane elastic constants C_{11} and C_{44}[4]. The experimental setup is shown in figure 2a. For the case where the wavelength is equal to or greater than the thickness of the specimen, the zero of the transmission coefficient resulting from the interference between the longitudinal and flexural modes is used to determine C_{11}. The maximum transmission is associated with the incidence angle for the flexural wave and is utilized to determine C_{44}. The experimental results for the 1-3 plane of the Cr_2O_3 coating attained using 2.0MHz tone-burst (fh=0.94 MHz·mm) are shown in figure 2b.

The transmission minimum angle is very sensitive to the value of the in-plane elastic constant C_{11} and insensitive to C_{44}. The maximum transmission coefficient is very sensitive to both C_{11} and C_{44}. To decouple their effect, the value of C_{11} is first calculated from the angle of transmission minimum and then C_{44} is determined from the maximum transmission angle.

Bulk Wave Velocity of the Cr_2O_3 Plate

As an alternative measuring technique and to determine additional elastic constants of the coating, the bulk wave velocity in the coating free plate was measured. We used the double-through-transmission self referential technique[5, 6] which tolerates imperfections in the sample geometry (as for ceramic coatings).

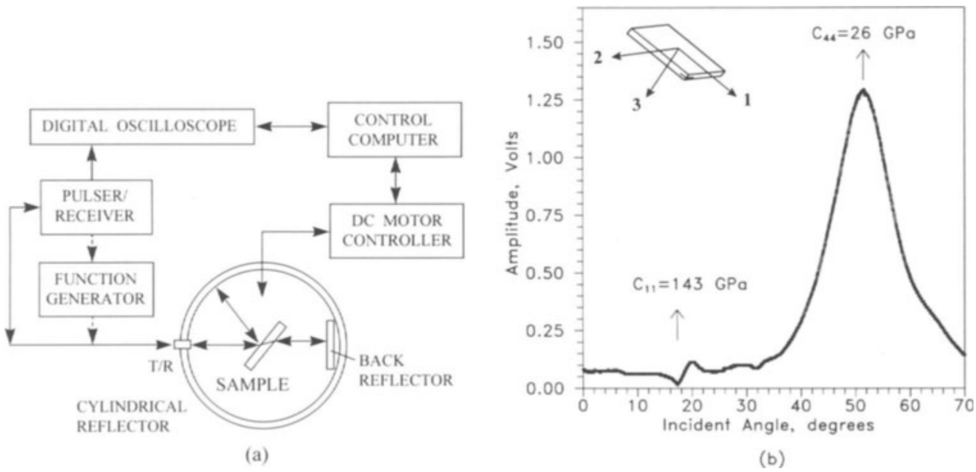

Figure 2. a) Experimental setup for measuring energy transmission and reflection amplitudes as a function of incident angle. b) Energy transmission coefficient versus incident angle for a Cr_2O_3 plate. C_{11} is determined from the transmission minimum, C_{44} from the transmission maximum location.

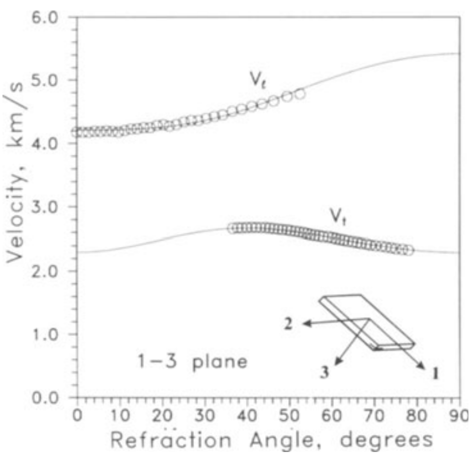

Figure 3. Longitudinal and transverse wave velocities versus refraction angle for 1-3 plane in plasma sprayed Cr_2O_3 plate. The sample orientation is shown in the figure.

Table 1. Elastic constants of plasma sprayed Cr_2O_3 as measured using ultrasonic techniques

Elastic Constant	Transmission/ reflection minima	1-2 plane	2-3 plane
C_{11}	143	144	161
C_{33}	86	86	87
C_{13}	50	40	43
C_{55}	26	25	26

It was impossible to measure the longitudinal velocity in Cr_2O_3 by the pulse overlap technique due to high attenuation, thus the half wavelength resonance was measured with a 5MHz transducer. The longitudinal velocity was found to be 4.14±0.04km/s.

The angle dependence of longitudinal and transverse bulk wave velocities are shown in figure 3. It can readily be seen that the coating is non-isotropic in the 1-3 plane. Similar results were obtained for the 2-3 plane. Table 1 summarizes the elastic constants of the Cr_2O_3 coating measured using the two methods described above.

Results for Cr_2O_3 coating on a substrate

The ultrasonic evaluation of the two-layer system, the coating on the substrate, was performed by measuring minima of the reflection coefficient at different incident angles. The experimental system used is shown in figure 2a. The amplitude of a double reflected ultrasonic signal versus incident angle was measured[7] using only one transducer. The trace velocity at a reflection minimum is expressed as $V_{tr}=V_f/\sin\theta$, where V_f is the velocity in the fluid[8].

Two cases were studied using the tone-burst technique: waves incident from the fluid on the sample from a) the coating side, and b) the substrate side.

THEORETICAL MODEL

To reconstruct the material parameters from the ultrasonic measurements and to evaluate the coating adhesion to the substrate, we use a theoretical model shown in figure 4a for ultrasonic wave interaction with the two-layer structure. Due to the nature of the plasma-spray process, the coating has multi-layered microstructure and is considered to be orthotropic. The substrate was assumed to be isotropic. Each layer is characterized by the appropriate thickness, density and elastic constants. The mechanical contact at the interface between the coating and substrate layers is simulated by longitudinal and transverse springs. Changing the spring constants represents different interfacial properties which affect the response of the structure and can later be compared to the experimental data. An assessment of the coating-substrate adhesion quality can thus be obtained.

A set of curves for energy reflection coefficients as a function of incident angle were calculated to simulate the ultrasonic measurements performed by the angle beam immersion technique. The calculations given in figure 4b are for the case of wave incidence from the fluid on the coating side of a two layer coating/substrate system.

Two sets of calculated data were compared and the trace velocities determined from a) the position of the reflection coefficient minimum and b) from the zero of the denominator of the transmission coefficient[8].

In accordance with the experimental procedure, two cases were considered: incidence from a) the coating side, and b) the substrate side.

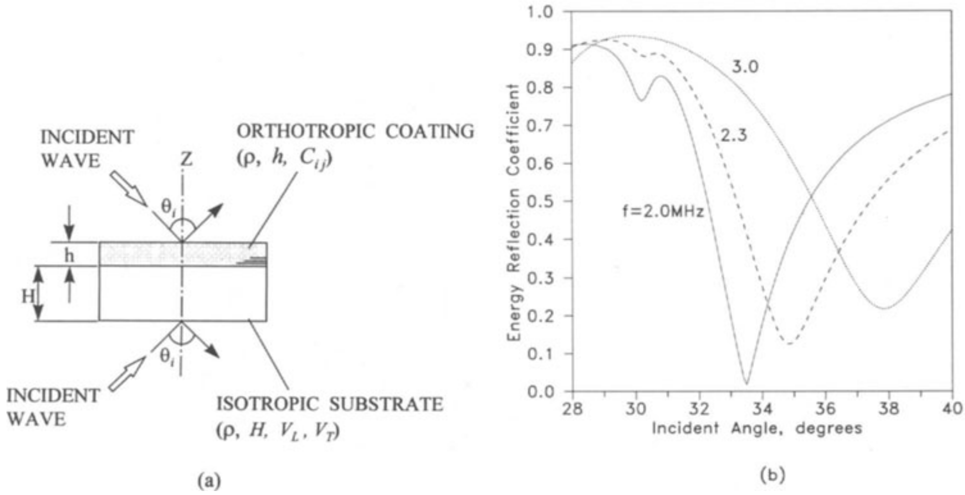

Figure 4. a) Schematic of the configuration used in the analysis. The coating is orthotropic and the substrate is isotropic. b) Energy reflection coefficient versus incident angle. Wave is incident from the coating side.

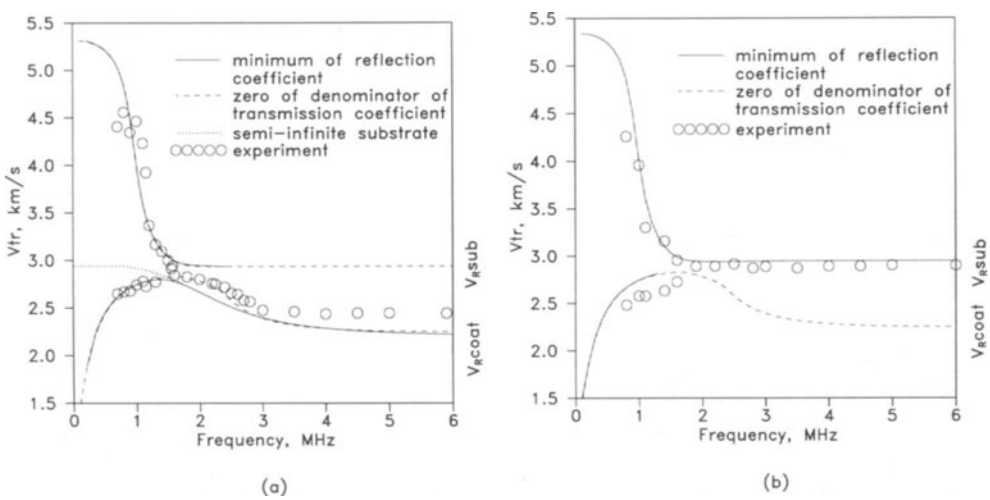

Figure 5. Trace velocities in Cr_2O_3-steel structure. a)wave incident from the coating side. b) wave incident from the substrate side

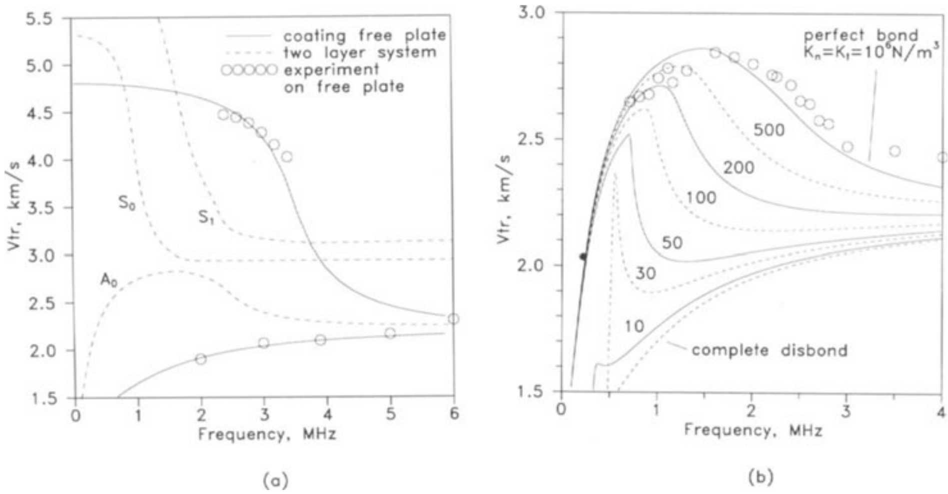

Figure 6. a) Comparison of the trace velocity data for the Cr_2O_3 plate (solid lines are theory, circles are experiment). b) Transition from poor to good bond demonstrated by various interfacial spring values (circles are experiment).

RESULTS AND DISCUSSION

The experimental results together with the prediction of the model are shown in figure 5. The ultrasonic reflectivity of the two layer system depends upon which side the incident waves come from (coating or substrate). At lower frequencies, flexural and longitudinal plate motions dominate the wave reflectivity. With frequency increase, Rayleigh wave behavior dominates of the reflection minimum. From figure 5a it is clear that the trace velocities calculated for the two layer system match the experimental data well and differ from the prediction for a coating on a semi-infinite substrate at low frequencies.

Effect of Interfacial Properties

The coating adhesion to the substrate is one of the most important characteristics of the plasma-spray process. To simulate its effect we used the spring boundary conditions between the coating and the substrate.

The theoretical trace velocities calculated from both the minima position of the energy reflection coefficient and the zeros of the denominator of the transmission coefficient are shown in figure 6. For a two layer system, it was found that both methods yielded similar results. The trace velocities for the two layer system (coating/substrate) and for the coating layer are shown in figure 6a where experimental points are shown only for the coating layer.

The bond quality is assessed by changing the stiffnesses of the springs. As demonstrated in figure 6b, near-zero stiffness will simulate the behavior of a free plate (single coating) while high stiffness will simulate a good bond. From comparing the theory and the experimental results for coating on a substrate one concludes that the bonding is good between the layers. Thus, good metallurgical contact is present in the specimen which was evaluated and this is supported by the micrograph shown in figure 1.

SUMMARY

This work is focused on ultrasonic characterization of Cr_2O_3 plasma sprayed ceramic coatings on a steel substrate. The coating elastic moduli, the porosity content and the coating adhesion to the substrate are important material properties necessary to measure for process optimization and to maintain product functionality in service.

To characterize the material fully, angle beam reflection spectroscopy has been performed on both coated samples and free coatings prepared by chemical dissolution of the steel substrate. It was found that the coatings are orthotropic and have significant porosity. To model ultrasonic wave interaction with the coated samples the two-layer, fluid loaded system is considered. The model utilizing an orthotropic coating on an isotropic substrate with spring boundary conditions is found to be adequate to describe the experimental data. The material properties are found using the least squares minimization of the error function between the experimental data and the model prediction.

The elastic properties of the coating were measured ultrasonically by different techniques. At low frequencies two modes are excited which degenerate into a Rayleigh mode at higher frequencies. The transition to the Rayleigh wave on the coating or the substrate occurs depending on the side of irradiation by the incident wave. The frequency at which this transition occurs depends upon the properties of both layers. Comparing theory and experiment for the coating/substrate two layer system makes it possible to retrieve information concerning the quality of the adhesion between the coating and the substrate. For the particular specimen studied, good adhesion is found between the coating and the substrate.

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REFERENCES

1. R.C. Tucker: in *Metals Handbook, Surface Engineering*, Vol. 5, C.M. Cottel, J.A. Sprague, F.A. Smidt, eds., ASM International, 1994, p. 497.
2. P. Fauchais: in *Engineering Materials Handbook, Ceramics and Glasses*, Vol. 4, S.J. Schneider, ed., ASM International, p. 202-209.
3. S.J. Schneider, ed., *Engineering Materials Handbook, Ceramics and Glasses*, Vol. 4, ASM International, p. 30.
4. S.I. Rokhlin, W. Wang: J. Acoust. Soc. Am., 94(5), November 1993, p. 2721.
5. Y.C. Chu, S.I. Rokhlin: J Acoust. Soc. Am., 95(6), June 1994, p. 3204.
6. S.I. Rokhlin, W. Wang: J Acoust. Soc. Am., 91(6), June 1992, p. 3303.
7. S.I. Rokhlin, W. Wang: J Acoust. Soc. Am., 86(5), November 1989, pp. 1876.
8. D. E. Chimenti, S.I. Rokhlin: J. Acoust. Soc. Am., 88(3), September 1990, p. 1603.